

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification⁵ : C09K 3/32, C02F 1/68 B01J 20/24	A1	(11) International Publication Number: WO 92/06146 (43) International Publication Date: 16 April 1992 (16.04.92)
(21) International Application Number: PCT/GB91/01711 (22) International Filing Date: 3 October 1991 (03.10.91) (30) Priority data: 9021509.6 3 October 1990 (03.10.90) GB (71)(72) Applicants and Inventors: ROBSON, David, James [GB/GB]; Lleiniau, Dwyran, Anglesey, Gwynedd LL61 6YG (GB). LAWYER, John, Mark [GB/GB]; 47 Bro Emrys, Tal-Y-Bont, Bangor, Gwynedd LL57 3YS (GB). HUGHES, Sara [GB/GB]; 4 Tan Y Bryn Terrace, Bangor, Gwynedd LL57 1HS (GB). (74) Agent: ATKINSON, Peter, Brich; Marks & Clerk, Suite 301, Sunlight House, Quay Street, Manchester M3 3FY (GB).		(81) Designated States: AT, AT (European patent), AU, BB, BE (European patent), BF (OAPI patent), BG, BJ (OAPI patent), BR, CA, CF (OAPI patent), CG (OAPI patent), CH, CH (European patent), CI (OAPI patent), CM (OAPI patent), CS, DE, DE (European patent), DK, DK (European patent), ES, ES (European patent), FI, FR (European patent), GA (OAPI patent), GB, GB (European patent), GN (OAPI patent), GR (European patent), HU, IT (European patent), JP, KP, KR, LK, LU, LU (European patent), MC, MG, ML (OAPI patent), MN, MR (OAPI patent), MW, NL, NL (European patent), NO, PL, RO, SD, SE, SE (European patent), SN (OAPI patent), SU ⁺ , TD (OAPI patent), TG (OAPI patent), US. Published <i>With international search report.</i>
(54) Title: METHOD OF ABSORBING HYDROPHOBIC WATER-IMMISCIBLE LIQUIDS (57) Abstract A method of absorbing hydrophilic water-immiscible liquids (e.g. oil) by using cellulosic plant material which has been modified by reaction of hydroxyl groups (e.g. by esterification) to render the material relatively more absorbent to hydrophobic liquids.		

PATENT COOPERATION TREATY

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

IDENTIFICATION OF THE INTERNATIONAL APPLICATION		Applicant's or Agent's File Reference																						
International Application No. PCT/GB 91/01711	PBA / SR / D08641 PWD																							
Receiving Office UK PATENT OFFICE	International Filing Date 3 OCTOBER 1991 (03.10.91)																							
Applicant (Name) ROBSON, DAVID J ET AL	Priority Date Claimed 3 OCTOBER 1990 (03.10.90)																							
BASIS OF REPORT																								
<p>1. AMENDMENTS AND/OR RECTIFICATIONS¹ — The amendments and/or rectifications made before this International Preliminary Examining Authority in respect of the claims, the description, and/or drawings in the above-identified international application are annexed to this report.</p> <p>a. <input checked="" type="checkbox"/> This report has been established on the basis of the following application documents:</p> <table style="width: 100%; border: none;"> <tr> <td style="width: 50%; vertical-align: top;"> <input checked="" type="checkbox"/> the application documents as filed </td> <td style="width: 50%;"></td> </tr> <tr> <td><input type="checkbox"/> description, pages</td> <td>as originally filed</td> </tr> <tr> <td>description, pages</td> <td>filed with your letter of</td> </tr> <tr> <td>description, pages</td> <td>filed with your letter of</td> </tr> <tr> <td>description, pages</td> <td>filed with your letter of</td> </tr> <tr> <td><input type="checkbox"/> claim(s)</td> <td>as originally filed</td> </tr> <tr> <td>claim(s)</td> <td>filed with your letter of</td> </tr> <tr> <td>claim(s)</td> <td>filed with your letter of</td> </tr> <tr> <td>claim(s)</td> <td>filed with your letter of</td> </tr> <tr> <td><input type="checkbox"/> drawings, sheet/fig.</td> <td>as originally filed</td> </tr> <tr> <td>drawings, sheet/fig.</td> <td>filed with your letter of</td> </tr> </table> <p>b. <input type="checkbox"/> The amendments resulted in the cancellation of the following sheets:</p> <p>c. <input type="checkbox"/> This report has been established as if the amendments indicated on the extra sheet have not been made, since, for the reasons indicated, they have been considered to go beyond the disclosure as filed.</p>			<input checked="" type="checkbox"/> the application documents as filed		<input type="checkbox"/> description, pages	as originally filed	description, pages	filed with your letter of	description, pages	filed with your letter of	description, pages	filed with your letter of	<input type="checkbox"/> claim(s)	as originally filed	claim(s)	filed with your letter of	claim(s)	filed with your letter of	claim(s)	filed with your letter of	<input type="checkbox"/> drawings, sheet/fig.	as originally filed	drawings, sheet/fig.	filed with your letter of
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<input type="checkbox"/> drawings, sheet/fig.	as originally filed																							
drawings, sheet/fig.	filed with your letter of																							
<p>2. PRIORITY²</p> <p>a. This report has been established as if no priority has been claimed due to the failure to furnish within the prescribed time limit the requested:</p> <p><input type="checkbox"/> copy of the earlier application whose priority has been claimed.</p> <p><input type="checkbox"/> translation of the earlier application whose priority has been claimed.</p> <p>b. <input type="checkbox"/> This report has been established as if no priority has been claimed due to the fact that the priority claim has been found invalid.</p> <p style="text-align: center;">Thus, for the purposes of this report, the international filing date indicated above is considered to be the relevant date.</p>																								
<p><small>¹ Where replacement sheets are annexed to this report, a translation of these replacement sheets must be furnished to the elected Offices within the time limit applicable under PCT Article 35(1).</small></p>																								

**PATENT COOPERATION TREATY
INTERNATIONAL PRELIMINARY EXAMINATION REPORT**

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CLASSIFICATION OF SUBJECT MATTER (If several classification symbols apply, indicate all.)

According to International Patent Classification (IPC) or to both National Classification and IPC

C09K 3/32, C02F 1/68 B01J 20/24

REASONED STATEMENT AS TO CLAIMS MEETING CRITERIA OF NOVELTY (N), INVENTIVE STEP (IS) AND INDUSTRIAL APPLICABILITY (IA)* AND CITATIONS* AND EXPLANATIONS* SUPPORTING SUCH STATEMENTCLAIM
NUMBERSTATEMENT
(CRITERIA)

CITATIONS AND EXPLANATIONS

1-24 YES(N, IS
IA)

All claims meet the requirement of industrial applicability.

All claims meet the requirements of novelty and inventive step since the method of absorbing hydrophobic water-immiscible liquids by treating the liquid with a modified material as claimed in Claims 1 and 18, an article comprising such a modified material, and a covering material as claimed in Claim 20, or a sheet of such material are neither disclosed nor suggested in the prior art cited on the International Search Report.

N N-WRITTEN DISCL SURES ⁹			
Kind of Non-Written Disclosure	Date of Written Disclosure referring to the Non-Written Disclosure	Date of Non-Written Disclosure	
CERTAIN PUBLISHED DOCUMENTS ¹⁰			
Application/Patent	Date of Publication	Filing Date	Priority Date (Valid Claim)
CERTAIN DEFECTS IN THE INTERNATIONAL APPLICATION ¹¹			
<p>The following defects in the form or contents of the international application have been noted.</p> <div style="height: 150px;"></div>			
CERTAIN OBSERVATIONS ON THE INTERNATIONAL APPLICATION ¹²			
<p>The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description have been noted.</p> <div style="height: 200px;"></div>			
CERTIFICATION			
Date Demand Submitted 5 MAY 1992 (05.05.92)		Date of Completion of the International Preliminary Examination Report 23 JUNE 1992 (23.06.92)	
International Preliminary Examining Authority UNITED KINGDOM PATENT OFFICE		Signature of Authorized Officer R HONEYWOOD	

22 Rec'd PCT/PTO 10 JUL 991

PCT/GB91/01711

PATENT COOPERATION TREATY

TO:

United States Patent
and Trademark Office
Washington, D.C.

FROM:

the INTERNATIONAL BUREAU of the
WORLD INTELLECTUAL PROPERTY
ORGANIZATION

NOTIFICATION CONCERNING
DOCUMENTS TRANSMITTED

Issued pursuant to PCT
Article 36(3)(a)

(as elected Office)

Date of Mailing:

30 June 1992 (30.06.92)

NOTIFICATION

The International Bureau transmits herewith the following documents
and number thereof:

1 (number of) copy(s) of the international preliminary
examination report (Article 36(3)(a)).

This notification is sent to the above addressee in its capacity as
an elected Office.

THE INTERNATIONAL BUREAU OF THE WORLD INTELLECTUAL PROPERTY ORGANIZATION

Mailing Address:

WIPO
34, chemin des Colombettes
1211 Geneva 20
Switzerland

Authorised Officer:

M. Abidine

MAY 1992

PATENT COOPERATION TREATY

	INTERNATIONAL APPLICATION NO. PCT/GB91/01711
NOTIFICATION OF ELECTION issued pursuant to PCT Rule 61.2	To: United States Patent and Trademark Office Washington, D.C. in its capacity as an elected Office
DATE OF MAILING OF THIS NOTIFICATION: 13 May 1992 (13.05.92)	From: The International Bureau of WIPO 1211 Geneva 20 Switzerland
APPLICANT (NAME): ROBSON, David, James et al.	
INTERNATIONAL FILING DATE: 03 October 1991 (03.10.91)	
PRIORITY DATE CLAIMED: 03 October 1990 (03.10.90)	
The said election was made in the demand received by the International Preliminary Examining Authority on: 05 May 1992 (05.05.92)	
 C. Combaz (Authorized Officer)	

PATENT COOPERATION TREATY

	INTERNATIONAL APPLICATION NO. PCT/GB91/01711
NOTIFICATION TO THE DESIGNATED OFFICE OF RECEIPT OF RECORD COPY issued under PCT Rule 24.2(a)	To: United States Patent and Trademark Office Washington, D.C.
APPLICANT'S OR AGENT'S FILE REFERENCE: PBA/SR/D086410PWO	in its capacity as a designated Office
DATE OF MAILING OF THIS NOTIFICATION: 25 October 1991 (25.10.91)	From: The International Bureau of WIPO 1211 Geneva 20 Switzerland
NAME(S) OF APPLICANT(S): ROBSON, David, J. et al.	
INTERNATIONAL FILING DATE: 03 October 1991 (03.10.91)	
PRIORITY DATE(S) CLAIMED: 03 October 1990 (03.10.90)	
DATE OF RECEIPT OF RECORD COPY BY INTERNATIONAL BUREAU: 24 October 1991 (24.10.91)	
C. Combaz (Authorized Officer)	

07 MAY 1992

**INTERNATIONAL APPLICATION
UNDER THE
PATENT COOPERATION TREATY
REQUEST**

THE UNDERSIGNED REQUESTS THAT THE PRESENT
INTERNATIONAL APPLICATION BE PROCESSED
ACCORDING TO THE PATENT COOPERATION TREATY

(The following is to be filled in by the receiving Officer)

INTERNATIONAL APPLICATION No. **PCT/GB 91 / 0 1 7 1 1**INTERNATIONAL FILING DATE: **03 10 91**
03 October 1991(Stamp) **United Kingdom Patent Office**
PCT International Application
Name of receiving Officer and "PCT International Application"Applicant's or agent's file reference **PBA/SR/D086410PWO**
(indicated by applicant if desired)

Box No. I TITLE OF INVENTION **IMPROVEMENTS IN OR RELATING TO THE**
ABSORPTION OF HYDROPHOBIC WATER-IMMISCIBLE LIQUIDS

Box No. II APPLICANT (WHETHER OR NOT ALSO INVENTOR); DESIGNATED STATES FOR WHICH HE/SHE/IT IS APPLICANT. Use this box for indicating the applicant or, if there are several applicants, one of them. If more than one person (includes, where applicable, a legal entity) is involved, continue in Box No. III.

The person identified in this box is (mark one check-box only):

☒ applicant and inventor*☐ applicant only

Name and address:** **ROBSON, DAVID JAMES**
LLEINIAU,
DWYRAN,
ANGLESEY,
GWYNEDD
LL61 6YG.
UNITED KINGDOM

Telephone number (including area code):

Telegraphic address:

Teleprinter address:

State of nationality:

UNITED KINGDOM

State of residence:*

UNITED KINGDOMThe person identified in this box is *applicant* for the purposes of (mark one check-box only):☒ all designated States☐ all designated States except the United States of America☐ the United States of America only☐ the States indicated in the "Supplemental Box"

Box No. III FURTHER APPLICANTS, IF ANY; (FURTHER) INVENTORS, IF ANY; DESIGNATED STATES FOR WHICH THEY ARE APPLICANTS (IF APPLICABLE). A separate sub-box has to be filled in in respect of each person (includes, where applicable, a legal entity). If the following two sub-boxes are insufficient, continue in the "Supplemental Box." (giving there for each additional person the same indications as those requested in the following two sub-boxes) or by using a "continuation sheet."

The person identified in this box is (mark one check-box only):

☒ applicant and inventor*☐ applicant only☐ inventor only*

Name and address:** **LAWTHER, JOHN MARK**
47 BRO EMRYS,
TAL-Y-BONT,
BANGOR,
GWYNEDD,
LL57 3YS. UNITED KINGDOM.

If the person identified in this sub-box is *applicant* (or *applicant and inventor*), indicate also:

State of nationality:

UNITED KINGDOM

State of residence:*

UNITED KINGDOMand whether that person is *applicant* for the purposes of (mark one check-box only):☒ all designated States☐ all designated States except the United States of America☐ the United States of America only☐ the States indicated in the "Supplemental Box"

The person identified in this sub-box is (mark one check-box only):

☒ applicant and inventor*☐ applicant only☐ inventor only*

Name and address:** **HUGHES, SARA,**
4 TAN Y BRYN TERRACE,
BANGOR,
GWYNEDD,
LL57 1HS.
UNITED KINGDOM

If the person identified in this sub-box is *applicant* (or *applicant and inventor*), indicate also:

State of nationality:

UNITED KINGDOM

State of residence:*

UNITED KINGDOMand whether that person is *applicant* for the purposes of (mark one check-box only):☒ all designated States☐ all designated States except the United States of America☐ the United States of America only☐ the States indicated in the "Supplemental Box"

* If the person indicated as "applicant and inventor" or as "inventor only" is not an *inventor* for the purposes of all the designated States, give the necessary indications in the "Supplemental Box."

** Indicate the name of a natural person by giving his/her family name first followed by the given name(s). Indicate the name of a legal entity by its full official designation. In the address, include both the postal code (if any) and the State (name).

*** If residence is not indicated, it will be assumed that the State of residence is the same as the State indicated in the address.

SUBSTITUTE SHEET

Box No. IV AGENT (IF ANY) OR COMMON REPRESENTATIVE (IF ANY); ADDRESS FOR NOTIFICATIONS (IN CERTAIN CASES). A common representative may be appointed only if there are several applicants and if no agent is or has been appointed; the common representative must be one of the applicants.

The following person (includes, where applicable, a legal entity) is hereby/has been appointed as agent or common representative to act on behalf of the applicant(s) before the competent International Authorities:

Name and address, including postal code and country:

If the space below is used instead for an address for notifications, mark here: ☐

ATKINSON, Peter Birch,
Marks & Clerk,
Suite 301, Sunlight House,
Quay Street,
Manchester,
M3 3JY.

United Kingdom

Telephone number (including area code):

061-832 9911

Telegraphic address: MARKLERK

MANCHESTER

Teleprinter address: 668953

MANPAT G

Box No. V DESIGNATION OF GROUPS OF STATES OR STATES⁽¹⁾; CHOICE OF CERTAIN KINDS OF PROTECTION OR TREATMENT. The following designations are hereby made (please mark the applicable check-boxes):

Regional Patent

☒ **EP European Patent⁽²⁾:** AT Austria, BE Belgium, CH and LI Switzerland and Liechtenstein, DE Germany, DK Denmark, ES Spain, FR France, GB United Kingdom, GR Greece, IT Italy, LU Luxembourg, NL Netherlands, SE Sweden, and any other State which is a Contracting State of the European Patent Convention and of the PCT

☒ **OA OAPI Patent:** Benin, Burkina Faso, Cameroon, Central African Republic, Chad, Congo, Gabon, Mali, Mauritania, Senegal, Togo, and any other State which is a Contracting State of OAPI and of the PCT; if other OAPI title desired, specify on dotted line⁽³⁾:

Côte d'Ivoire, Guinea

National Patent (if other kind of protection or treatment desired, specify on dotted line⁽³⁾)

<input checked="" type="checkbox"/> AT Austria ⁽³⁾	<input checked="" type="checkbox"/> KR Republic of Korea ⁽³⁾
<input checked="" type="checkbox"/> AU Australia ⁽³⁾	<input checked="" type="checkbox"/> LK Sri Lanka
<input checked="" type="checkbox"/> BB Barbados	<input checked="" type="checkbox"/> LU Luxembourg ⁽³⁾
<input checked="" type="checkbox"/> BG Bulgaria ⁽³⁾	<input checked="" type="checkbox"/> MC Monaco ⁽³⁾
<input checked="" type="checkbox"/> BR Brazil ⁽³⁾	<input checked="" type="checkbox"/> MG Madagascar
<input checked="" type="checkbox"/> CA Canada	<input checked="" type="checkbox"/> MW Malawi ⁽³⁾
<input checked="" type="checkbox"/> CH and LI Switzerland and Liechtenstein	<input checked="" type="checkbox"/> NL Netherlands
<input checked="" type="checkbox"/> DE Germany ⁽³⁾	<input checked="" type="checkbox"/> NO Norway
<input checked="" type="checkbox"/> DK Denmark	<input checked="" type="checkbox"/> PL Poland ⁽³⁾
<input checked="" type="checkbox"/> ES Spain ⁽³⁾	<input checked="" type="checkbox"/> RO Romania
<input checked="" type="checkbox"/> FI Finland	<input checked="" type="checkbox"/> SD Sudan
<input checked="" type="checkbox"/> GB United Kingdom	<input checked="" type="checkbox"/> SE Sweden
<input checked="" type="checkbox"/> HU Hungary	<input checked="" type="checkbox"/> SU Soviet Union ⁽³⁾
<input checked="" type="checkbox"/> JP Japan ⁽³⁾	
<input checked="" type="checkbox"/> KP Democratic People's Republic of Korea ⁽³⁾	<input checked="" type="checkbox"/> US United States of America ⁽³⁾

Space reserved for designating States (for the purposes of a national patent) which have become party to the PCT after the issuance of this sheet:

Mongolia, Czechoslovakia

⁽¹⁾ The applicant's choice of the order of designations may be indicated by marking the check-boxes with sequential arabic numerals (see also the "Notes to Box No. V").

⁽²⁾ The selection of particular States for a European patent can be made upon entering the national (regional) phase before the European Patent Office (see also the "Notes to Box No. V").

⁽³⁾ If another kind of protection or a title of addition or, in the United States of America, treatment as a continuation or a continuation-in-part is desired, specify according to the instructions given in the "Notes to Box No. V".

Box No. VI PRIORITY CLAIM (IF ANY). The priority of the following earlier application(s) is hereby claimed:

Country (country in which it was filed if national application; one of the countries for which it was filed if regional or international application)	Filing Date (day, month, year)	Application No.	Office of filing (fill in only if the earlier application is an international application or a regional application)
UNITED KINGDOM (1)	03-OCTOBER-90 03. 10. 90	9021509 . 6	
(2)			
(3)			

(Letter codes may be used to indicate country and/or Office of filing)

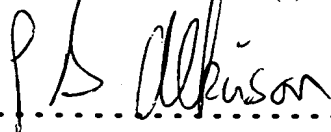
When the earlier application was filed with the Office which, for the purposes of the present international application, is the receiving Office, the applicant may, *against payment of the required fee*, ask the following:☒ the receiving Office is hereby requested to prepare and transmit to the International Bureau a certified copy of the above-mentioned earlier application/of the earlier applications identified above by the numbers (insert the applicable numbers).....**Box No. VII EARLIER SEARCH (IF ANY).** Fill in where a search (international, international-type or other) by the International Searching Authority has already been requested (or completed) and the said Authority is now requested to base the international search, to the extent possible, on the results of the said earlier search. Identify such search either by reference to the relevant application (or the translation thereof) or by reference to the search request.

International application number or number and country (or regional Office) of other application:

International/regional/national filing date:

Date of request for search:

Number (if available) given to search request:

Box No. VIII SIGNATURE OF APPLICANT(S) OR AGENT

ATKINSON, PETER BIRCH

If the present Request form is signed on behalf of any applicant by an agent, a separate power of attorney, appointing the agent and signed by the applicant is required. If in such case it is desired to make use of a general power of attorney (deposited with the receiving Office), a copy thereof must be attached to this form.

Box No. IX CHECK LIST (To be filled in by the Applicant)

This international application contains the following number of sheets:

- | | | |
|----------------|-----------|---------------|
| 1. request | 3 | sheets |
| 2. description | 9 | sheets |
| 3. claims | 2 | sheets |
| 4. abstract | 1 | sheets |
| 5. drawings | 2 | sheets |
| Total | 17 | sheets |

Figure number of the drawings (if any) is suggested to accompany the abstract for publication.

This international application as filed is accompanied by the items marked below:

- ☐ separate signed power of attorney
- ☐ copy of general power of attorney
- ☐ priority document(s) (see Box No. VI)
- ☐ receipt of the fees paid or revenue stamps
- ☐ cheque for the payment of fees
- ☐ request to charge deposit account
- ☐ other document (specify)

(The following is to be filled in by the receiving Office)

1. Date of actual receipt of the purported international application: **03 October 1991 03 10 91**

2. Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application:

3. Date of timely receipt of the required corrections under Article 11 of the PCT:

4. Drawings ☐ Received ☐ No Drawings

(The following is to be filled in by the International Bureau)

Date of receipt of the record copy:

24 OCTOBER 1991**(24. 10. 91)**



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification⁵ : C09K 3/32, C02F 1/68 B01J 20/24	A1	(11) International Publication Number: WO 92/06146 (43) International Publication Date: 16 April 1992 (16.04.92)
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(54) Title: METHOD OF ABSORBING HYDROPHOBIC WATER-IMMISCIBLE LIQUIDS (57) Abstract <p>A method of absorbing hydrophilic water-immiscible liquids (e.g. oil) by using cellulosic plant material which has been modified by reaction of hydroxyl groups (e.g. by esterification) to render the material relatively more absorbent to hydrophobic liquids.</p>		

METHOD OF ABSORBING HYDROPHOBIC WATER-IMMISCIBLE LIQUIDS

The present invention relates to the absorption of hydrophobic water-immiscible liquids, particularly but not exclusively for cleaning-up liquid spillages as well as to materials and products for effecting the absorption of hydrophobic water-immiscible liquids.

A wide variety of hydrophobic water-immiscible liquids absorbents have been used in the past to clean up liquid spillage in water and from around machinery (particularly to clean up oil spillage). These absorbents fall into two main categories: firstly synthetic polymers (e.g. polypropylene) and secondly natural plant based materials such as straw, peat and sawdust. However, both of these categories of materials have their disadvantages. In particular, the synthetic polymers (e.g. polypropylene) are relatively expensive in comparison with plant material whereas plant material itself has a combination of hydrophilic and hydrophobic properties and attracts both water and hydrophobic water-immiscible liquids. An additional disadvantage of these natural products is that they deteriorate during storage.

A further prior proposal made by Midland Silicones (part of DOW Corning) is the use of silanated sawdust to render the sawdust more hydrophobic and more attractive to hydrophobic water-immiscible liquids. The silanated sawdust was particularly developed to absorb oil. This proposal has the disadvantage that silanation is a relatively expensive chemical modification.

A further proposal based on the use of natural products is disclosed in EP-A-0 094 363 which describes a plant material based product specifically as an oil absorbing composition which comprises at least 50% by weight of hydrophobic cellulose pulp blended with 30%-50% of an organic cellulose paper pulp filler. The cellulose pulp is rendered hydrophobic by a conventional non-bonding hydrophobing treatment including inter alia a conventional sizing treatment. However, a disadvantage of the oil absorbing products disclosed in EP-A-0 094 363 is that they only float on water for a limited time.

It is an object of the present invention to obviate or mitigate the abovementioned disadvantages.

According to a first aspect of the present invention there is

provided, for use as an absorbent of hydrophobic water-immiscible liquids, cellulosic plant material which has been rendered relatively more attractive to hydrophobic water-immiscible liquids by chemical reaction of hydroxyl groups in the plant material.

For convenience the plant material which has been rendered relatively more attractive to hydrophobic water-immiscible liquids by said chemical reaction is referred to hereinafter as the "modified plant material". The term "plant material" as used herein covers "raw" plant material (possibly formed into a product such as a paper or sheet, e.g. a TMP paper), as well as products obtained by processing of the plant material (e.g. conventionally produced papers).

According to a second aspect of the invention there is provided a method of absorbing hydrophobic water-immiscible liquids comprising treating the liquid with the modified plant material.

A third aspect of the invention provides an article for absorbing hydrophobic water-immiscible liquids comprising the modified plant material within a covering material through which liquid may pass.

A fourth aspect of the invention comprises the modified plant material in sheet form.

Examples of hydrophobic water-immiscible liquids which may be absorbed by the modified plant material are crude and refined oil, solvents such as white spirit, toluene benzene and pesticide residues.

The preferred forms of plant material for use in the invention comprise lignocellulose, which is the collective name given to lignin, cellulose, and hemicellulose. It is however also possible to use plant material which contains neither lignin nor hemicellulose (e.g. cotton).

The preferred modification treatment for the plant material is esterification. If the plant material contains only cellulose then it is the cellulose which will be esterified. If the plant material is lignocellulose then the phenolic hydroxyl groups of the lignin and possibly also the hydroxyl groups of the cellulose and the hemicellulose are esterified. Preferably the acid residues in the ester groups are of the formula Alk-C(O)-O in which Alk is an alkyl group of 1-4 carbon atoms. Processes for esterifying cellulose and lignocellulose in the plant material are already known (see for example EP-A-0 213 252) and such prior processes are suitable for producing modified plant material for use in this invention. Typically the

esterification will be effected by treating the plant material with the corresponding anhydride, removing excess anhydride, and then heating the plant material in an oven, eg in the temperature range 90-150°C. The degree of esterification should be sufficient to render the plant material more attractive to hydrophobic water-immiscible liquids whilst still retaining internal hydrogen bonding to maintain the integrity of the material. Usually, the degree of esterification will be such as to provide a 5-40% weight gain for the plant material. Preferably the weight gain is in the range 12-25%.

The preferred method of esterification is by acetylation since acetic anhydride which is of relatively low cost may be used as the acetylating agent.

Treatments other than esterification which may be used for introducing hydrophobic groups into the plant material and rendering it relatively more attractive to hydrophobic water-immiscible liquids. Such treatments include reaction with an isocyanate so as to convert the hydroxyl groups of the plant material to urethane linkages. Examples of suitable isocyanates are monoisocyanates such as propyl isocyanate, butyl isocyanate and octadecyl isocyanate.

The agent used for rendering the plant material more attractive to water-immiscible liquids may be of di-or higher functionality so as to provide a degree of cross-linking.

The preferred plant material used as starting material for modification treatment is lignocellulose in the form of thermomechanically pulped fibre (preferably unbleached) comprising bundles of 3-5 cells so that the individual fibres have a length up to 5mm. Other examples of plant material which may be used include chips, plant stem segments, whole plant stems. Sources of plant material for modification treatment include wood, straw, flax, linseed, bagasse, sisal, jute, kenaf, miscanthus, coir, cotton and hemp.

Different plant materials do vary in their lignin content, e.g. cotton has no lignin whereas wood has approximately 30%. For the present invention it is preferred to use a plant material that has not previously been delignified for reasons of cost and enhanced reactivity.

The modified plant materials are eminently suitable for absorption of hydrophobic water-immiscible liquids. In particular,

they are capable of absorbing up to 50 times their own weight of hydrophobic water-immiscible liquids from a spillage thereon in water and will retain up to 30 times their own weight when removed from water and allowed to drain. There is the additional advantage that the modified plant material forms a discrete mass of hydrophobic water-immiscible liquid and plant material which floats on clean water whereas untreated plant material forms a mass of hydrophobic water-immiscible liquid and plant material which floats on an emulsified hydrophobic liquid/water mixture. In other words, use of untreated material causes oil to be "dragged" into the water whereas the modified material leaves the water "clean". This has considerable implications for the clean-up of hydrophobic water-immiscible spillages in inland waterways or in areas where environmental protection is important. The chemically modified plant material has the further advantage, over untreated fibres, in that it is less biodegradable and therefore less likely to deteriorate during storage.

The modified plant material may be presented for use in the absorption of hydrophobic water-immiscible liquids in a number of different forms. For example, the modified material may be contained within an outer "covering" through which the liquid may pass. Such a "covering" could for example be a net or porous sheet. It is therefore possible to provide the modified material in the form of a boom or pillow, i.e. a form in which oil absorbents are commonly used for cleaning oil spillages in water. To clean-up an oil-in-water spillage the boom or pillow is simply drawn through the water. Alternatively particulate or fibrous modified material may be spread on to the water surface by dropping from an aircraft. Alternatively, particulate or fibrous modified material can be blown on to the water surface.

It is also possible to provide the modified material in sheet form (eg a paper or fabric). Thus for example a sheet of lignocellulose material may be rendered attractive to hydrophobic water-immiscible liquids by any of the abovementioned treatments (eg acetylation) and used for cleaning up spillages of hydrophobic water-immiscible liquids, particularly spillages from machines or vehicles.

It should however be appreciated that the use of the modified plant material is not restricted to the clean-up of spillages. The

modified material could for example be used in filters designed to separate and recover hydrophobic water-immiscible liquids from hydrophobic water-immiscible liquid and water mixtures. Alternatively, modified lignocellulose could be used to retain hydrophobic liquids on lignocellulose in moist or wet conditions, for example, for improving the retention of transformer oils on electrical papers and increasing the intervals between oil replacement caused by moisture ingress.

The invention is illustrated by the following non-limiting Examples together with Figs.1 and 2 of the accompanying drawings which are plots of data obtained in Example 2.

Example 1

Production of Acetylated Lignocellulose

The method used was similar to that described in Example VII of EP-A-0 2213 262. Spruce thermomechanical fibre pulp (TMP) was dipped for 1 minute in liquid acetic anhydride. Excess anhydride was squeezed out from the pulp by applying mechanical compression forces to the material yielding an acetic anhydride to fibre pulp ratio of 2.5 w/w. The impregnated pulp was then heated at 120° C for different times. After the reaction a vacuum was applied. The fibre pulp was then air conditioned. The results are shown in Table 1.

Table 1

Sample	Reaction Time at 120° C (hours)	Weight gain due to acetylation (%)
Spruce TMP	0.5	11.7
	1.0	22.4
	2.0	24.8
	4.0	36.5

Example 2

Removal of Oil from Sea-Water (Laboratory Test)

This Example demonstrates the removal of Medium Fuel Oil (MFO) and Transformer Oil (Class 1 uninhibited mineral insulating oil) using

modified lignocellulose. The modified lignocellulose used was thermo-mechanically pulped wood fibre acetylated in accordance with the method described in Example 1 above and with an acetyl weight gain of between 17% and 20%, and the sea-water was collected from the Irish Sea off Anglesey.

The method used was an adaptation of that described in Example 1 of EP-A-0 094 363. Briefly this adapted method was as follows: 5gms of the modified lignocellulose fibre were mixed with 200 ml of medium fuel oil or transformer oil, and 200 ml of water. The mixture was stirred for 5 minutes using a magnetic stirrer. The mixture was allowed to stand for 5 minutes then poured through a screen. The liquid-containing absorption agent collected on the screen and was allowed to drain for 5 minutes. The procedure was repeated using untreated fibres.

The liquid-containing fibre was weighed and the ratio of liquid to fibre weight calculated. The results are shown in Table 2. Additionally, the oil/water mixture recovered from the screening process was analysed to determine the relative proportions of the oil and water. From this information relative weights of the fibre, oil and water in the liquid containing fibre was calculated. The results obtained for MFO are shown in Fig. 1 and those for Transformer Oil are shown in Fig. 2.

Table 2

OIL/WATER WEIGHT GAIN BY FIBRES
(multiple of initial fibre weight)

	MFO	Transformer Oil
ACETYLATED FIBRE	28.1	20.3
UNTREATED FIBRE	30.8	22.4

As shown in Table 2 the overall weight gain by untreated and acetylated fibres was similar.

It was however found that the oil uptake of the acetylated fibres was greater than for the untreated fibre, as seen by reference to the drawings.

Fig. 1a illustrates the results obtained using untreated fibre whereas Fig. 1b shows the results for acetylated fibre. A comparison

of Figs. 1a and 1b shows that the liquid absorbed by the acetylated fibres comprised a greater percentage of MFO than the untreated fibres. More particularly, about 70% of the liquid absorbed by the treated fibre was MFO in comparison to about 60% as absorbed by the untreated fibres. It will be seen from Fig. 2 that the difference was more substantial in the case of transformer oil for which the liquid absorbed by the acetylated fibres (Fig. 2b) comprised above 80% oil in comparison with a figure of about 45% for the untreated fibres (Fig. 2 a).

Example 3

Flotation trials in sea-water.

Acetylated fibre as used in Example 2 and untreated fibre were separately floated on sea-water. Untreated fibre sank completely in sea-water after 2 days. Acetylated fibre started to sink in sea-water after 5 days. A slowly reducing proportion of the acetylated fibre continued to float for up to a month.

Acetylated fibre which had previously absorbed Medium Fuel Oil floated on sea-water for at least two months.

Example 4

Effect on water quality.

The effect of the absorption of oil by the untreated and acetylated fibres on water quality below the main fibre mass was assessed using Methods 1 and 2 below. The acetylated fibres used as in Example 2.

Method 1. Extraction and Weight Measurement.

Fibres were mixed with oil and sea water in the ratio 5 gm fibres:100 ml Medium Fuel Oil:100 ml sea-water. A water sample from below the main fibre/oil mass was taken in a 100 ml syringe. The oil was extracted from the sea-water by mixing with 100 ml of chloroform. Sea-water was used as a control. The chloroform was separated from the water and samples of the chloroform evaporated. The weight of the remaining residue, expressed as a percentage of the chloroform sample, was:

untreated fibre	0.098%
acetylated fibre	0.006%
sea-water control	0.010%

Approximately 1.5% of the 100 ml of Medium Fuel Oil stirred into the untreated fibre/oil/seawater mixture is emulsified by the untreated fibres. A significantly lower amount was emulsified by the acetylated fibres.

Method 2. Extraction and UV Absorption.

UV peak absorbance of the chloroform extract obtained in Method 1 was 100 times greater for the extract from the untreated fibre mixture than for the extract from the acetylated fibre mixture. Water recovered from screening 200 ml Transformer Oil, 200 ml water and 5 gm fibre showed a UV peak absorbance over 25 times greater from the untreated fibre mixture than from the acetylated fibre mixture.

Example 5

Removal of White Spirit from Deionised Water.

The method was adapted from Example 1 of EP-A-0 094 363. Briefly this adapted method was as follows: 5 gms of acetylated fibre (acetyl weight gain 14-17%) were mixed with 200 ml of white spirit and 200 ml of deionised water. The mixture was stirred for 5 minutes using a rotary stirrer. The mixture was allowed to stand for 5 minutes and then poured through a screen. The liquid containing absorption agent collected on the screen was allowed to drain for 5 minutes. The liquid containing fibre was weighed and the ratio of liquid to fibre weight calculated, and the results are shown in Table 3.

TABLE 3

WHITE SPIRIT/WATER WEIGHT GAIN BY FIBRES

(multiple of initial fibre weight)

ACETYLATED FIBRE	15.7
UNTREATED FIBRE	17.9

The remaining white spirit and water which had not been

absorbed by the fibre was collected, separated and the water and white spirit fractions weighed. From the weights of these fractions the uptake of the water by the fibre and the uptake of white spirit by the fibre were calculated. These values (expressed as a percentage of the total uptake) are shown below in Table 4.

	<u>TABLE 4</u>	
	WATER UPTAKE	WHITE SPIRIT UPTAKE
	(%)	(%)
ACETYLATED FIBRE	19	81
UNTREATED FIBRE	63	37

CLAIMS

1. A method of absorbing hydrophobic water-immiscible liquids comprising treating the liquid with cellulosic plant material which has been modified to render it relatively more absorbent to hydrophobic water-immiscible liquids by chemical reaction of hydroxyl groups in the lignocellulose material.
2. A method as claimed in claim 1 wherein the modification is esterification.
3. A method as claimed in claim 2 wherein the esterified material has a weight gain of 5-40% as compared to the unesterified material.
4. A method as claimed in claim 3 wherein the weight gain is 12 to 25%.
5. A method as claimed in any one of claims 2 to 4 wherein the acid residues in the esterified material are of the formula Alk-C(O)-O in which Alk is an alkyl group of 1 to 4 carbon atoms.
6. A method as claimed in claim 5 wherein the esterification is acetylation.
7. A method as claimed in any one of claims 1 to 6 wherein the plant material comprises lignocellulose.
8. A method as claimed in claim 7 wherein the lignocellulose is thermomechanically pulped fibre.
9. A method as claimed in claim 7 wherein the lignocellulose comprises plant material chips, plant stem segments and/or whole plant stems.
10. A method as claimed in any one of claims 1 to 6 wherein the source of the lignocellulose is selected from wood, straw, flax, linseed, bagasse, sisal, jute, kenaf, miscanthus, coir, cotton and hemp.
11. A method as claimed in any one of claims 1 to 10 wherein the water-immiscible liquid is an oil.
12. A method as claimed in claim 11 wherein the oil is an oil spillage in water.
13. A method as claimed in claim 12 wherein the modified plant material is spread on to the surface of the water.
14. A method as claimed in claim 12 which comprises drawing through the oil an article which comprises the modified plant material within an outer covering through which the oil may pass.

15. A method as claimed in any one of claims 1 to 10 wherein the water-immiscible liquid is an organic solvent or a pesticide residue.

16. A method as claimed in any one of claims 1 to 10 for the filtration or removal of a hydrophobic water-immiscible liquid from a mixture of such a liquid with water.

17. A method as claimed in any one of claims 1 to 10 for the retention of transformer oil on a paper.

18. A method of absorbing hydrophobic water-immiscible liquids comprising treating the liquid with lignocellulose material which has been modified to render it relatively more absorbent to hydrophobic water-immiscible liquids by chemical reaction of hydroxyl groups in the lignocellulose material.

19. The use as an absorbent of hydrophobic water-immiscible liquids of cellulosic plant material which has been rendered relatively more absorbent to hydrophobic water-immiscible liquids by chemical reaction of hydroxyl groups in the lignocellulose material.

20. An article for absorbing hydrophobic water-immiscible liquids comprising cellulosic plant material which has been rendered relatively more attractive to hydrophobic water-immiscible liquids by chemical reaction of hydroxyl groups in the plant material, and a covering material through which the hydrophobic liquid may pass provided around the modified plant material.

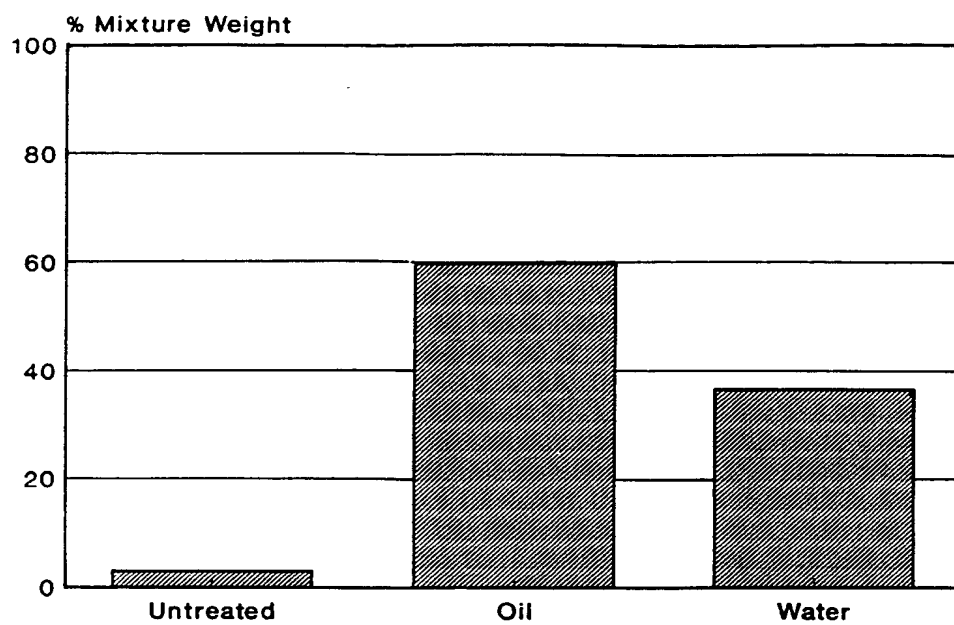
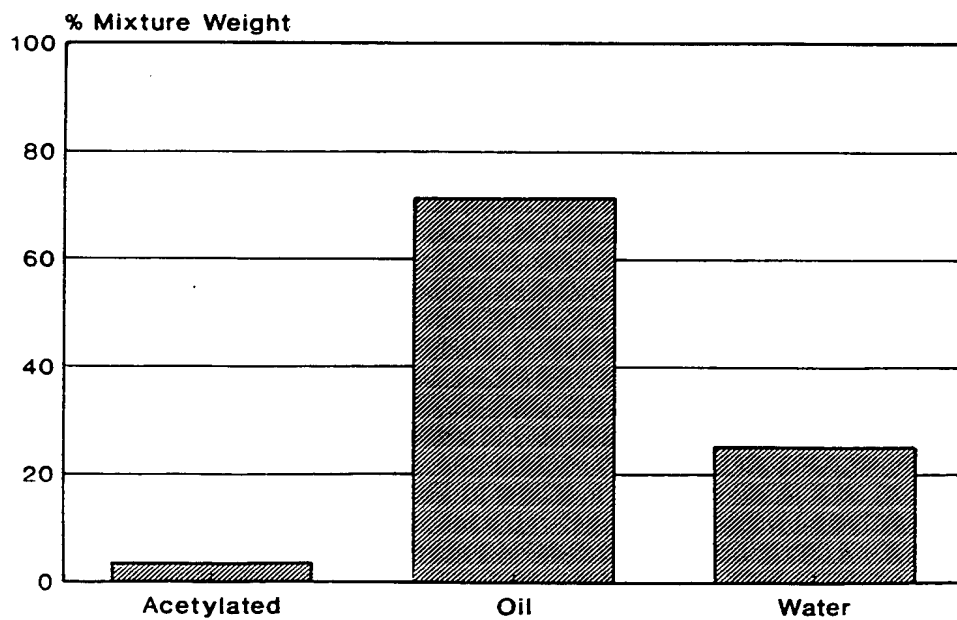
21. An article as claimed in claim 20 in the form of a boom or pillow.

22. A sheet of cellulosic plant material which has been rendered relatively more attractive to hydrophobic water-immiscible liquids by chemical reaction of hydroxyl groups in the plant material.

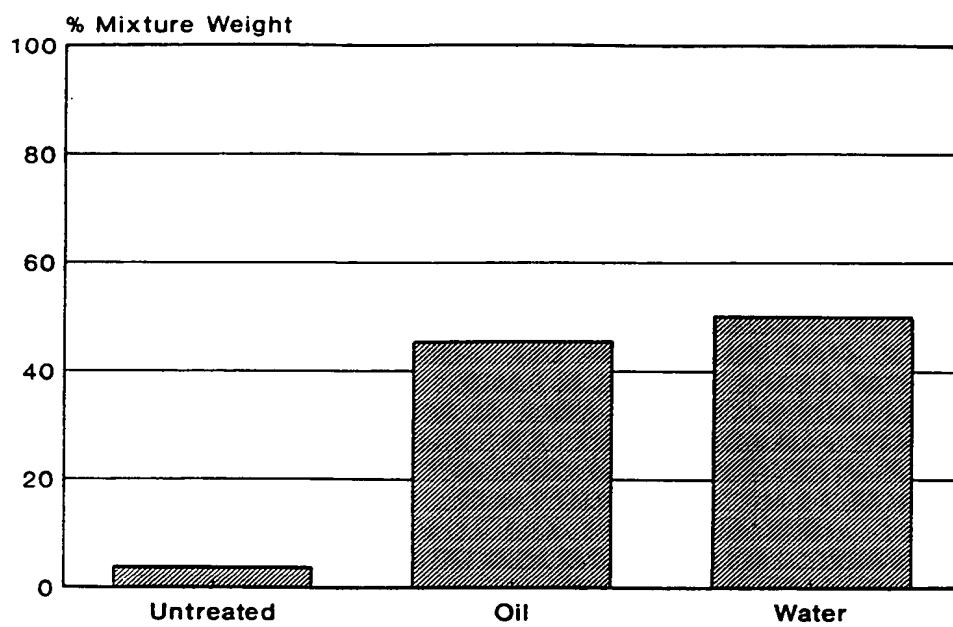
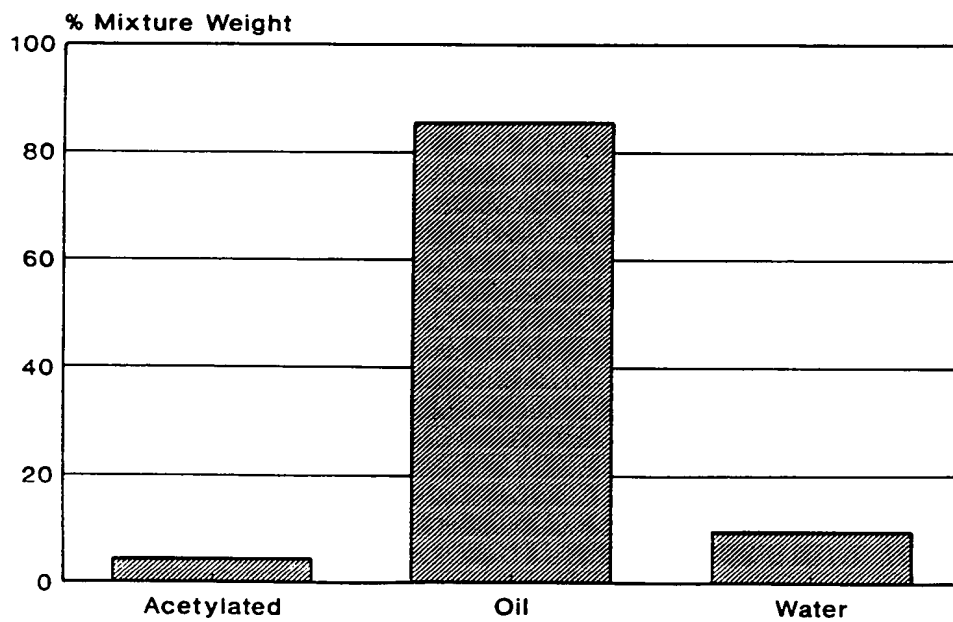
23. An article as claimed in claim 20 or 21 or a sheet as claimed in claim 22 wherein the plant material comprises lignocellulose.

24. An article or sheet as claimed in claim 23 comprising acetylated lignocellulose.

1 / 2

FIG 1aFIG 1b

2 / 2

FIG 2aFIG 2b

INTERNATIONAL SEARCH REPORT

PCT/GB 1/01711

International Application

I. CLASSIFICATION F SUBJECT MATTER (if several classification symbols apply, indicate all)⁶

According to International Patent Classification (IPC) or to both National Classification and IPC

Int.Cl. 5 C09K3/32; C02F1/68; B01J20/24

II. FIELDS SEARCHED

Minimum Documentation Searched⁷

Classification System

Classification Symbols

Int.Cl. 5

C09K ;

C02F ;

B01J

Documentation Searched other than Minimum Documentation
to the Extent that such Documents are Included in the Fields Searched⁸III. DOCUMENTS CONSIDERED TO BE RELEVANT⁹

Category ¹⁰	Citation of Document, ¹¹ with indication, where appropriate, of the relevant passages ¹²	Relevant to Claim No. ¹³
Y	US,A,4 925 343 (RAIBLE) 15 May 1990 see column 4, line 53 - line 68; claims 1,6,7,8 ---	1-13, 18-20
Y /	FR,A,2 466 564 (CTTN) 10 April 1981 see page 1, line 35 - page 2, line 13; claims 1,2 ---	1-13, 18-20
A	US,A,4 605 640 (FANTA) 12 August 1986 see column 2, line 16 - column 3, line 32; claims 1-5 ---	1-24
A /	WO,A,8 810 183 (WEYERHAEUSER) 29 December 1988 see page 4, line 13 - page 5, line 3; claims 1,2 ---	1-24
A /	EP,A,0 094 363 (PAPYRUS KOPPARFORS) 16 November 1983 see claims 1,2,3 ---	1-24
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¹⁰ Special categories of cited documents :

- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier document but published on or after the international filing date
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- "O" document referring to an oral disclosure, use, exhibition or other means
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"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

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IV. CERTIFICATION

Date of the Actual Completion of the International Search

Date of Mailing of this International Search Report

16 DECEMBER 1991

7. 11. 92

International Searching Authority

Signature of Authorized Officer

EUROPEAN PATENT OFFICE

NICOLAS H.J.F.

III. DOCUMENTS CONSIDERED TO BE RELEVANT

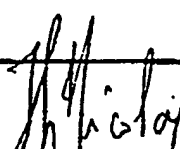
(CONTINUED FROM THE SECOND SHEET)

Category °	Citation of Document, with indication, where appropriate, of the relevant passages	Relevant to Claim No.
A	EP,A,0 002 070 (FISCHER) 30 May 1979 see abstract; claims 1,2,3,5 ---	1-24
A	DE,A,3 423 885 (MEYNEN) 31 January 1985 see the whole document ---	1-24

**ANNEX TO THE INTERNATIONAL SEARCH REPORT
ON INTERNATIONAL PATENT APPLICATION NO. GB 9101711
SA 51855**

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report. The members are as contained in the European Patent Office EDP file on
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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US-A-4925343	15-05-90	EP-A- 0414382	27-02-91
FR-A-2466564	10-04-81	None	
US-A-4605640	12-08-86	JP-A- 62043483	25-02-87
WO-A-8810183	29-12-88	AU-A- 2081888	19-01-89
EP-A-0094363	16-11-83	AU-A- 1436583	17-11-83
		CA-A- 1208621	29-07-86
		JP-A- 58210979	08-12-83
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		JP-A- 54095991	28-07-79
DE-A-3423885	31-01-85	None	

23 APR 93 11:54 AM & CERN 100 001 001 International Application		
I. CLASSIFICATION OF SUBJECT MATTER (If several classification symbols apply, indicate all)		
According to International Patent Classification (IPC) or to both National Classification and IPC Int.Cl. 5 C09K3/32; C02F1/68; B01J20/24		
II. FIELDS SEARCHED		
Minimum Documentation Searched?		
Classification System	Classification Symbols	
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Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched ³		
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Y	US,A,4 925 343 (RAIBLE) 15 May 1990 see column 4, line 53 - line 68; claims 1,6,7,8	1-13, 18-20
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A	WO,A,8 810 183 (WEYERHAEUSER) 29 December 1988 see page 4, line 13 - page 5, line 3; claims 1,2	1-24
A	EP,A,0 094 363 (PAPYRUS KOPPARFORS) 16 November 1983 see claims 1,2,3	1-24
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¹⁰ Special categories of cited documents: ^{"A"} document defining the general state of the art which is not considered to be of particular relevance ^{"E"} earlier document but published on or after the international filing date ^{"L"} document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) ^{"O"} document referring to an oral disclosure, use, exhibition or other means ^{"P"} document published prior to the international filing date but later than the priority date claimed ^{"T"} later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention ^{"X"} document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step ^{"Y"} document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art. ^{"A"} document member of the same patent family		
IV. CERTIFICATION		
Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report	
16 DECEMBER 1991	7. 11. 92	
International Searching Authority	Signature of Authorized Officer	
EUROPEAN PATENT OFFICE	NICOLAS H.J.F. 	

III. D DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)

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A	EP, A, 0 002 070 (FISCHER) 30 May 1979 see abstract; claims 1, 2, 3, 5 ---	1-24
A	DE, A, 3 423 885 (MEYNEN) 31 January 1985 see the whole document ---	1-24

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FR-A-2466564	10-04-81	None	
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		CA-A- 1208621	29-07-86
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		JP-A- 54095991	28-07-79
DE-A-3423885	31-01-85	None	

Page ...

CLASSIFICATION OF SUBJECT MATTER (If several classification symbols apply, indicate all.)		
According to International Patent Classification (IPC) or to both National Classification and IPC		
C09K 3/32, C02F 1/68 B01J 20/24		
REASONED STATEMENT AS TO CLAIMS MEETING CRITERIA OF NOVELTY (N), INVENTIVE STEP (IS) AND INDUSTRIAL APPLICABILITY (IA) AND CITATIONS AND EXPLANATIONS SUPPORTING SUCH STATEMENT		
CLAIM NUMBER	STATEMENT (CRITERIA)	CITATIONS AND EXPLANATIONS
1-24	YES(N, IS IA)	<p>All claims meet the requirement of industrial applicability.</p> <p>All claims meet the requirements of novelty and inventive step since the method of absorbing hydrophobic water-immiscible liquids by treating the liquid with a modified material as claimed in Claims 1 and 18, an article comprising such a modified material and a covering material as claimed in Claim 20, or a sheet of such material are neither disclosed nor suggested in the prior art cited on the International Search Report.</p>

02 MAR 1992
08/0500.0

See notes on accompanying sheet

INTERNATIONAL SEARCH REPORT

International Application No

PCT/GB 91/01711

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all)⁶

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Int.Cl. 5 C09K3/32; C02F1/68; B01J20/24

II. FIELDS SEARCHEDMinimum Documentation Searched⁷

Classification System

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Documentation Searched other than Minimum Documentation
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A	US,A,4 605 640 (FANTA) 12 August 1986 see column 2, line 16 - column 3, line 32; claims 1-5 ---	1-24
A	WO,A,8 810 183 (WEYERHAEUSER) 29 December 1988 see page 4, line 13 - page 5, line 3; claims 1,2 ---	1-24
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¹⁰ Special categories of cited documents:^{"A"} document defining the general state of the art which is not considered to be of particular relevance^{"E"} earlier document but published on or after the international filing date^{"L"} document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)^{"O"} document referring to an oral disclosure, use, exhibition or other means^{"P"} document published prior to the international filing date but later than the priority date claimed^{"T"} later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention^{"X"} document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step^{"Y"} document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.^{"&"} document member of the same patent family**IV. CERTIFICATION**

Date of the Actual Completion of the International Search

16 DECEMBER 1991

Date of Mailing of this International Search Report

17. 11. 92

International Searching Authority

EUROPEAN PATENT OFFICE

Signature of Authorized Officer

NICOLAS H.J.F.

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)

Category °	Citation of Document, with indication, where appropriate, of the relevant passages	Relevant to Claim No.
A	EP,A,0 002 070 (FISCHER) 30 May 1979 see abstract; claims 1,2,3,5 ---	1-24
A	DE,A,3 423 885 (MEYNEN) 31 January 1985 see the whole document ---	1-24

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